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TECHNICAL REPORT ARCCB-TR-92036

**DEGRADATION AND FAILURE MODES OF
CARBON/BISMALEIMIDE LAMINATES
SUBJECTED TO A TROPICAL EXPOSURE**

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13. ABSTRACT (Maximum 200 words) A scanning electron microscope (SEM) was used to characterize the degradation of the outermost layers of carbon/bismaleimide laminates as the result of exposure to a natural tropical environment. The surface that was exposed to the sun for 4000 hours suffered complete degradation of the bismaleimide matrix to a shallow depth. SEM fractography showed little difference in the fracture appearance of the fibers in a carbon/bismaleimide laminate, which displayed considerable fiber pullout compared to fibers in a carbon/epoxy laminate, which displayed little pullout.				
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INTRODUCTION

Recent work by Underwood characterized the flexural strength and fracture toughness behavior of carbon/epoxy and carbon/bismaleimide laminates and related some of the behavior to micromechanisms as revealed by scanning electron fractography (refs 1,2). In the recent work, two of the more significant effects noted were the following. First, the J-integral fracture toughness of carbon/bismaleimide was found to decrease following exposure to a natural tropical environment. A second and separate finding, not related to environmental concerns, was that the fracture of carbon/bismaleimide occurred by extensive fiber pullout and gave high fracture toughness ($\approx 80 \text{ MPa m}^{1/2}$ for 0/90 layups), whereas fracture of carbon/epoxy showed little pullout and low fracture toughness ($\approx 20 \text{ MPa m}^{1/2}$). Each of these findings raised questions and suggested further investigation. With regard to environmental effects, Johnson (ref 3) suggested a study of the relationships between the microcharacteristics of degraded surfaces and the subsequent measurements of fracture properties. Regarding fracture in general as related to fiber pullout, we wondered if the important pullout process, associated with high fracture toughness, is also associated with a different fiber fracture mechanism from that for fibers which do not pull out.

The preceding questions involve the interrelation of microfailure mechanisms with macromechanical behavior. The objective here is to use the key tool for studying micromechanisms, a scanning electron microscope (SEM), to investigate these questions--in the one case, the relationship between environmental surface degradation and fracture properties, and in the other, the relationship between fiber failure mode and the high toughness, fiber pullout behavior.

SURFACE DEGRADATION

In the prior work (refs 1,2), the tropical environment of northern Australia was applied for 4000 hours to [(0/90)₂/0] and [(0₂/90)₂/0₂] layups of Fiberite X-86 tape, with Celanese G-40 and G-50 fibers used for 0 and 90-degree plies, respectively. There was no significant moisture absorption at the end of the exposure, but a powdery residue formed on the top (sun facing) surface, and subsequent tests showed indications of degraded fracture properties, as outlined in forthcoming paragraphs.

Surface degradation was characterized here by utilizing replica techniques in addition to direct observation of the sample surface. Replicas were made by wetting one side of a 0.2-mm thick acetate tape with acetone, pressing it onto the surface of interest, and allowing it to resolidify. The sample side of the replica was sputter-coated with palladium to impart the electrical conductivity required for 20 keV SEM studies. In addition to providing a high fidelity representation of topographical features, this type of replica tends to extract, *in situ*, mechanically-bonded surface material; chemically-bonded material will not be affected. Therefore, the replicas will reveal a measure of surface damage perhaps not fully appreciated by directly observing the sample surface.

Figures 1 and 2 are SEM photos from unloaded areas of the surface of [(0₂/90)₂/0₂] carbon/bismaleimide samples exposed in the tropics and tested for fracture toughness and flexural strength. Figure 1 contrasts the top and bottom surfaces of an exposed specimen. The top surface was exposed for the full 4000 hours to sunlight as well as moisture, whereas the bottom surface was subjected to moisture without sunlight. It is clear that the bismaleimide matrix has been completely degraded and removed from the top layer of fibers on the top surface, while only isolated patches of degradation have occurred on the bottom surface.

Figure 2 compares replicas of the top and bottom surfaces of the exposed sample of Figure 1. The bottom surface shows only a few isolated carbon fibers with associated crevices, in marked contrast to the broom-like appearance of the top surface. The top surface shows nearly complete degradation and separation of the matrix from the fibers. The stripping process is expected to remove loose and partially bonded matrix particles but not properly bonded matrix. Therefore, the bare fibers in Figure 2b, with the

longitudinal texture apparent, show that the degradation was quite complete.

The surface degradation characterized by Figures 1 and 2 should be considered in relation to the fracture toughness and flexural strength results of the prior work (ref 2). Table 1 lists results pertinent to the discussion. Center-notched tensile panels were used for fracture toughness tests, and four-point bend tests were used for flexural strength tests (ref 2); three replicates of each test were done. The most significant effect of the tropical exposure on the test results in Table 1 is with the J-integral based fracture toughness. The K value corresponding to applied J at the maximum load point of the test, K_{Jmax} , was used in this case (ref 2) to obtain a measure of fracture resistance, which included the considerable permanent deformation sustained by the specimens. The area under the load-deflection curve was extensive for the unexposed samples, but was considerably reduced by the exposure, as indicated by the lower K_{Jmax} values. It appears that the primary effect of the surface degradation from tropical exposure is to lower the resistance of carbon/bismaleimide to sustain permanent deformation before fracture.

Table 1. Fracture Toughness and Flexural Strength of $[(0_2/90)_3/0_2]$ Carbon/Bismaleimide Laminates

Exposure	Fracture Toughness, MPa m ^{1/2}		Flexural Strength, MPa
	K_{max}	K_{Jmax}	
none	100; 88; 90	484; 417; 375	498; 443; 518
tropical	91; 89; 89	410; 320; 254	549; 461; 409

FIBER FAILURE MODE

Figures 3 and 4 address the question of the failure mode of the individual carbon fibers, as affected by the presence or absence of fiber pullout in the two types of material. The figures show normal and oblique views at high magnification of the fracture surface from fracture toughness tests of the two materials. Figure 3 shows a minimum amount of fiber/matrix separation in the carbon/epoxy, compared to the much more extensive fiber/matrix separation and concomitant fiber pullout observed in the carbon/bismaleimide; arrows show examples of both features. No significant difference in failure mode of the fibers is apparent. The oblique views of Figure 4 give the same basic result: significant pullout differences but no significant fiber failure mode differences in the two materials.

SUMMARY

Complete degradation of the extreme outer layer of carbon/bismaleimide laminates exposed to the tropical sun has been shown by scanning electron microscopy. The degradation is apparently shallow enough in these 2-mm thick laminates to affect only the resistance to large permanent deformation of the laminates during the final fracture process. The area under the load-deflection curve and the critical K values corresponding to J at maximum load are significantly reduced by the environmental degradation of $[(0_2/90)_3/0_2]$ laminates. These results show that caution is advised for (a) applications with long outdoor exposures or exposures combined with mechanical abrasion that could accelerate the degradation; and (b) applications in which the permanent deformation of the laminate is relied upon in a fail-safe design.

Scanning electron fractography has shown that the failure mode of individual carbon fibers in the epoxy and bismaleimide matrix laminates is not significantly different. This is in contrast to the significant difference in fiber pullout behavior of the two materials, with little pullout in carbon/epoxy and extensive pullout in carbon/bismaleimide. This gives further support to the belief (ref 2) that the pullout process

itself is the major energy dissipative process in the fracture of these laminates. The pullout absorbs the energy and thereby increases the fracture toughness, and the final failure of the fiber contributes little to this useful process of energy dissipation.

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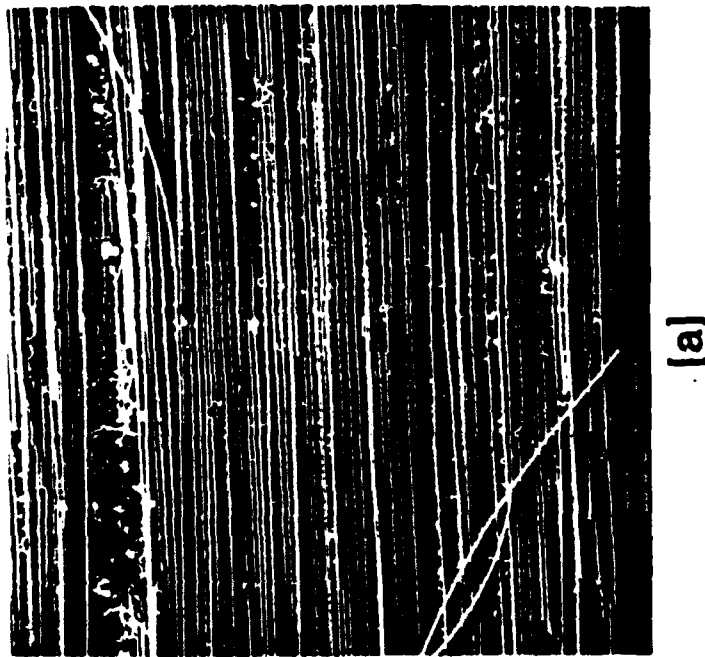


Figure 1. Appearance of top and bottom surfaces following exposure, 0,90 carbon/bismaleimide (100X):
(a) exposed, top; (b) exposed, bottom.

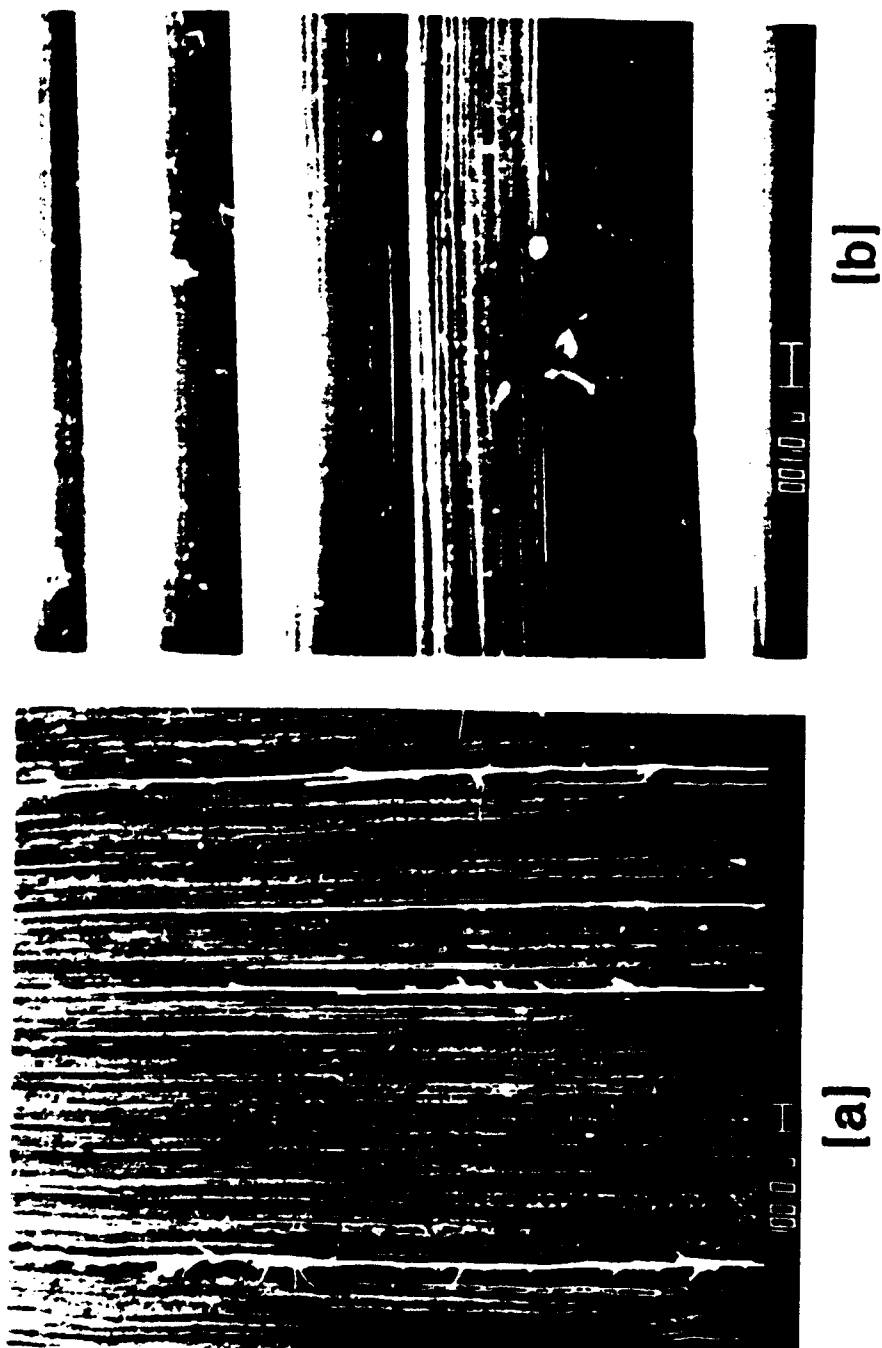


Figure 2. Replicas of top and bottom surfaces; 0,90 carbon/bismaleimide:
 (a) bottom surface (30X); (b) exposed, top (5000X).

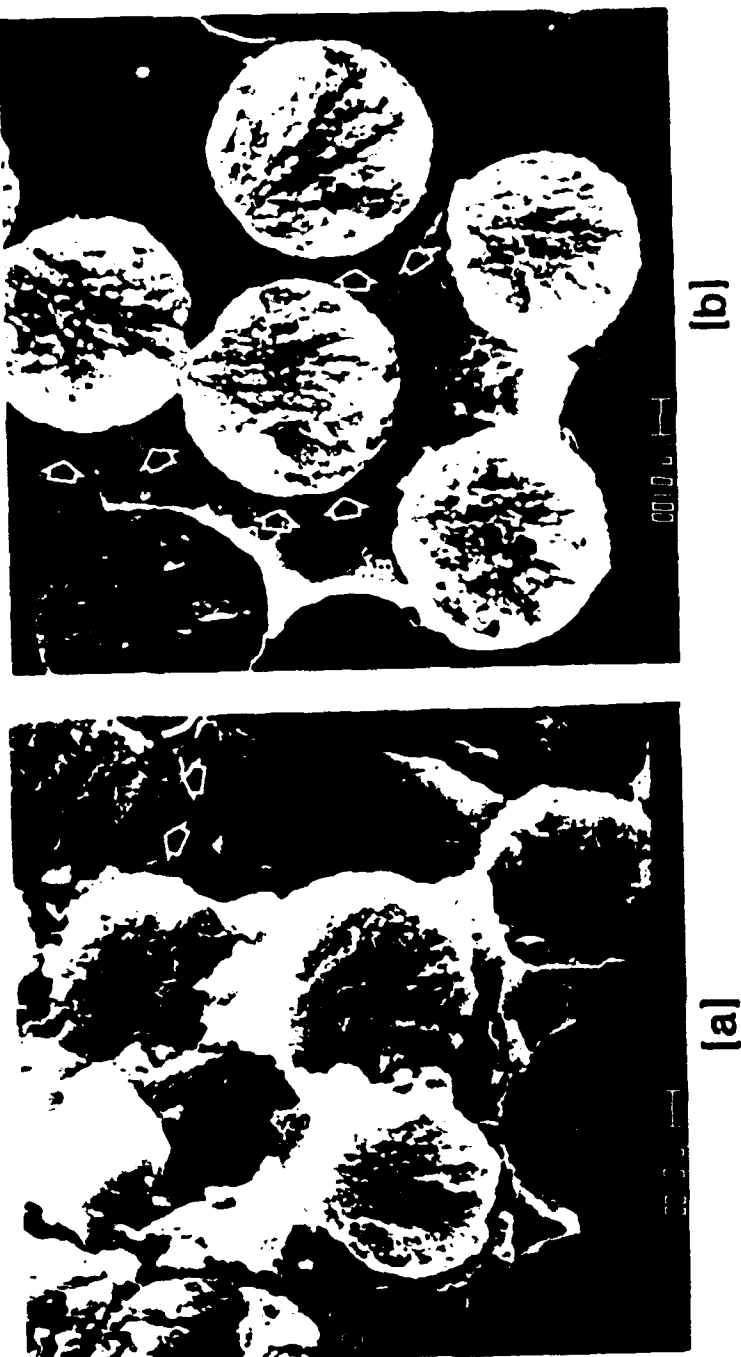


Figure 3. Normal view of fracture surfaces of 0/90 carbon/epoxy and carbon/bismaleimide (5000X):
(a) carbon/epoxy; (b) carbon/bismaleimide.

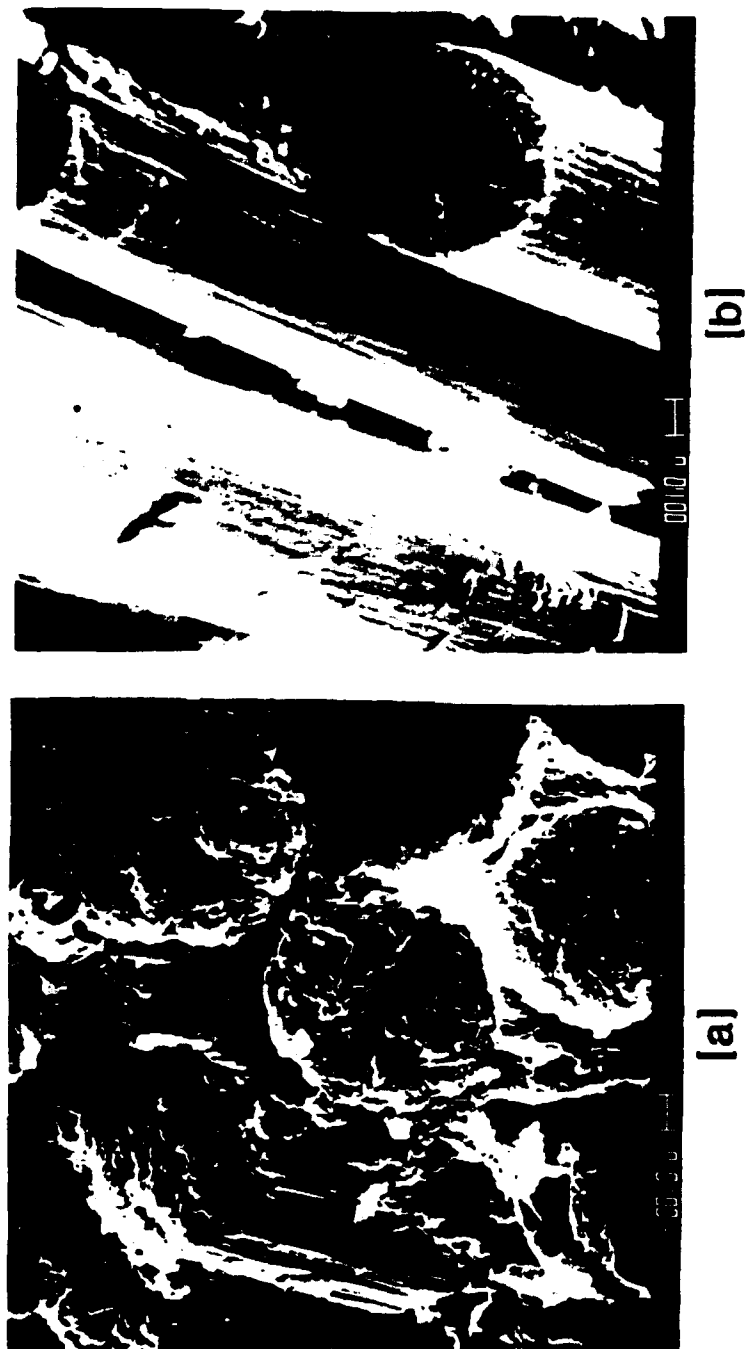


Figure 4. Oblique view of fracture surfaces of 0/90 carbon/epoxy and carbon/bismaleimide (5000X):
(a) carbon/epoxy; (b) carbon/bismaleimide.

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